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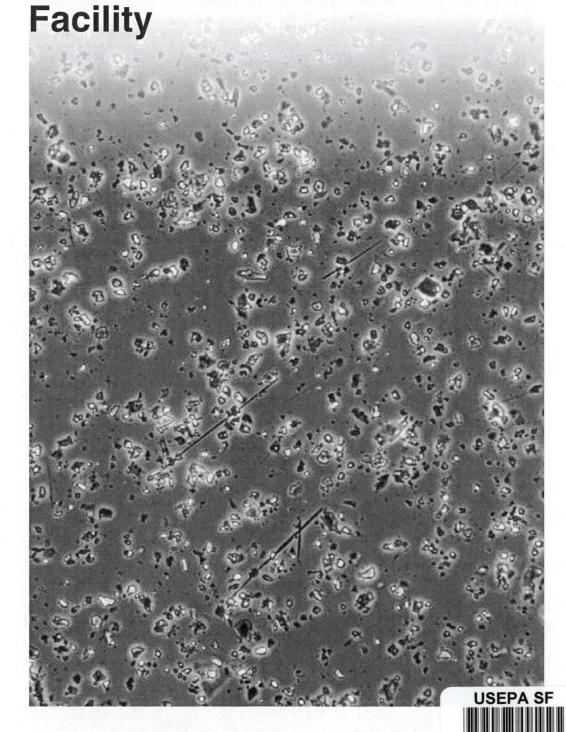
Office of Environmental Assessmen

Investigation and Engineering Unit

March 2004



Study of Asbestos
Contamination of Former
Vermiculite Northwest / W.R.
Grace Vermiculite Exfoliation



## Study of Asbestos Contamination of Former Vermiculite Northwest / W.R. Grace Vermiculite Exfoliation Facility

March 2004

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### **Abstract**

The Environmental Protection Agency (EPA) has determined that vermiculite ore mined at the former W.R. Grace vermiculite mine in Libby, Montana, is contaminated with asbestos. Libby ore was processed at vermiculite exfoliation plants across the country including one located in Spokane, Washington. EPA Region 10 conducted a three phase study at the Spokane vermiculite exfoliation plant to determine if asbestos fibers in the soil at the site could become airborne when the soil was disturbed. First, soil samples were taken from several locations within the site boundary and analyzed using polarized light microscopy and X-ray diffraction. Analysis revealed that most of the asbestos in the soil is similar to the amphibole asbestos that occurs in vermiculite from Libby, Montana.

In phase two, twelve soil specimens were collected from the site and eleven were agitated inside a laboratory enclosure equipped with air monitoring equipment. Ten of the soil specimens contained asbestos that became airborne when the soil was agitated. Filters used for collection of air samples were analyzed with a transmission electron microscope (TEM) and were found to contain asbestos. The concentration of asbestos found in the air ranged from 0.051 fibers per cubic centimeter (f/cc) to 10.713 f/cc.

During phase three, EPA staff collected eighteen air samples while performing property maintenance and excavation tasks at two locations on-site. Personal monitors with air filters were placed in the workers breathing zone and stationary monitors were placed near the work areas. Some filters were analyzed using phase contrast microscopy (PCM). Six of these samples contained fibrous material measuring from 0.02 f/cc to 0.25 f/cc of air. Other samples were analyzed using TEM. Four of these samples contained asbestos fiber concentrations ranging from 0.010 f/cc to 0.045 f/cc of air. Several asbestos fibers were also detected in filters from stationary air monitors.

Analysis of air samples by TEM showed a majority of the fibers were amphiboles similar to those that occur in vermiculite from Libby, Montana. Approximately 52% of the airborne asbestos fibers detected in both experiments had aspect ratios of greater than or equal to 20:1 and approximately 15% of the fibers counted were greater than or equal to 20 µm in average length.

This study clearly shows that asbestos in the soil at the former vermiculite exfoliation plant in Spokane can be released into the air when the soil is actively disturbed. Because there is a clear pathway for asbestos to move from contaminated soil to the air, individuals working on the site can be exposed to potentially hazardous levels of airborne asbestos fibers.

## Acknowledgements

Many people contributed to the quality of this study. We would like to thank our colleagues at EPA Region 10 including Earl Liverman; Julie Wroble; Bruce Woods, Ph.D.; David Terpening; Marion "Doc" Thompson; Grechen Schmidt; and Christopher Moffett.

We are grateful to those who provided analytical support: David Frank, PhD., EPA Region 10 for analyzing samples by x-ray diffraction; Susan Davis, Washington State Department of Ecology, for analyzing our samples by polarized light microscopy and phase contrast microscopy; and John Harris, MPH, and his staff at Lab/Cor, Inc., for analyzing our samples by transmission electron microscopy.

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### Introduction

The former vermiculite exfoliation plant in Spokane, Washington, received vermiculite ore from Libby, Montana. In 2000 and 2001 the EPA Region 10 Office of Environmental Cleanup (ECL) confirmed the presence of asbestos in the soil at this site. The following year investigators with the EPA Region 10 Office of Environmental Assessment (OEA) were asked to determine whether asbestos present in the soil at the site could become airborne when the soil was disturbed, posing a potential exposure risk to people working on the site. The study also attempted to further characterize the types of asbestos fibers present.

The OEA study was conducted between May and October 2002 and consisted three phases:

- general surveillance of the site and collection of soil samples;
- air monitoring in a laboratory enclosure while soil samples from the site were agitated;
- air monitoring on site while property maintenance and excavation tasks were performed.

This report provides background information on Libby vermiculite and health impacts from asbestos. These are followed by the a description of the study, the analytical results and a brief description of potential risk. The OEA study was conducted under the authority of the Comprehensive Environmental Response, Compensation and Liability Act.

## **Background**

## Regulation of Asbestos

The Environmental Protection Agency (EPA) and the Occupational Safety and Health Administration (OSHA) are the two primary federal agencies that have promulgated regulations designed to reduce potential exposure to asbestos in the environment and the workplace. These two agencies share common definitions of asbestos, but differ on how asbestos contamination is measured and what response actions are required.

EPA and OSHA currently regulate only six forms of asbestos: chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite. All are members of the amphibole group of minerals with the exception of chrysotile, which is a member of the serpentine group of minerals. The EPA regulates asbestos under the Clean Air Act (CAA), the Toxic Substances Control Act (TSCA), and the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA). For the purposes of this report, the term asbestos will be used to describe fibrous minerals, some of which are not specifically included in the CAA or TSCA, but are subject to regulation under CERCLA.

CERCLA and Chapter 40 of the Code of Federal Regulations (CFR) allow EPA to abate, prevent, minimize, stabilize, mitigate, or eliminate any release or threat of release of hazardous substances or pollutants and contaminants where such release results in a threat to public health or welfare of the United States or the environment. Among the criteria used to determine if a threat exists include:

- actual or potential exposure to nearby populations from hazardous substances or pollutants and contaminants;
- high levels of hazardous substances or pollutants or contaminants in soils at or near the surface that may migrate;
- weather conditions, such as wind, that may cause hazardous substances or pollutants and contaminants to migrate or be released; or
- other situations or factors that may pose threats to public health or welfare of the United States or the environment.

# Minerals Associated with Libby Vermiculite

Studies of Libby vermiculite have shown the ore contains varying quantities of amphibole minerals, which have commonly been referred to as asbestos. A United States Geological Survey (USGS) study identified several amphiboles including winchite, richterite, tremolite, magnesioriebeckite, magnesio-arfvedsonite, and possibly edenite in Libby vermiculite. The most abundant of these were winchite, richterite, and tremolite. The USGS also determined the morphology of a majority of the amphiboles in their study falls between prismatic crystals and asbestiform fibers. All of the amphiboles observed, with the possible exception of magnesioriebeckite, can occur in the fibrous or asbestiform habit.<sup>2</sup> Among the list of amphiboles present in Libby vermiculite, only tremolite is included by name in EPA's regulations.<sup>3</sup>

anthophyllite; tremolite; and actinolite.

Deer, W.A., Howie, R.A., and Zussman, 1992, An Introduction to Rock Forming Minerals, Second Edition, pages 223-275 and 344-352, Pearson Education Limited, Essex, England.

Meeker, G.P., Bern, A.D., Brownfield, I.K., Lowers, H.A., Sutley, S.J., Hoefen, T.M., and Vance, J.S., The Composition and Morphology of Amphiboles from the Rainy Creek Complex, Near Libby, Montana. American Mineralogist, Volume 88, pages 1955-1969, 2003.
 Title 40 Code of Federal Regulations, part 763. Asbestos means the asbestiform varieties of: Chrysotile; crocidolite; amosite;

## Health Studies Involving Amphibole Asbestos

Asbestos is a Class A human carcinogen that commonly enters the body through inhalation. Inhalation of airborne asbestos fibers increases the risk of nonmalignant interstitial and pleural lung disease, lung cancer, and mesothelioma. Several studies have implicated fibrous amphiboles, such as those found in Libby vermiculite, in cases of asbestos-related disease.

The National Institute of Occupational Health (NIOSH) studies show former Libby mine workers had substantial occupational exposure to asbestos. These studies documented significantly increased rates of asbestosis and lung cancer among former workers. Additional asbestosrelated disease was also reported among the household members of former mine workers and by other Libby residents who had no known direct connection with the mining operation.<sup>5</sup>

EPA Region 8 has been engaged in a long-term site investigation at the former W.R. Grace vermiculite mine in Libby, MT. The investigation has generated new information about asbestos contamination in the vermiculite ore from that mine. EPA Region 8 has determined that an imminent and substantial endangerment to public health exists in Libby due to asbestos contamination.<sup>6</sup>

Health impacts of exposure to the asbestos contaminated Libby vermiculite were identified by W.R. Grace as early as 1977. Internal correspondence from the W.R. Grace Construction Products Division showed ex-employees at Libby experienced lung cancer at a rate five times the national average. This conclusion was based on an in-house study conducted by W.R. Grace. The correspondence stated "...we have experienced asbestosis in 41.5% of the workers (with over 10 years' service) in Libby, as well as in 28% of the workers (with over 10 years' service) exposed to Libby ore in the expanding plants."

Other facilities which processed Libby vermiculite ore are reporting health impacts. A recently published case, reported in the American Journal of Respiratory and Critical Care Medicine, described the results of analyzing lung tissue of a former employee at a vermiculite exfoliation plant in California. The worker was exposed to the same fibrous amphiboles as those found in Libby while working two summers at the California exfoliation plant. This was the worker's only identified exposure to asbestos during his career. The worker died of asbestosis fifty years later.<sup>8</sup>

The American Journal of Industrial Medicine reported on a case involving an individual who worked for the Air Force, between 1970 and 1987, packing cans of paint for shipment two or three hours per day. The cans were packed in boxes which he filled with vermiculite. He did not wear a respirator. He knew of no asbestos exposure in the workplace or home environment. He has been diagnosed with a probable case of asbestosis.<sup>9</sup>

A 2003 study of another non-regulated amphibole implicated the amphibole fluoro-edenite in a cluster of deaths from pleural mesothelioma. These studies provide evidence that mineral forms of asbestos not specifically regulated by EPA or OSHA can cause diseases such as asbestosis, lung cancer and mesothelioma. The risk of developing these diseases depends upon many factors, including the chemistry and shape of the fiber, level of exposure, duration of exposure, the individual's physiological response to fiber exposure, and the smoking history of the exposed individual.

## Vermiculite Processing

Vermiculite is produced from ore mined throughout the world. Processed vermiculite has many desirable properties. It is absorbent, light weight, and fire resistant and has numerous uses. Historically, vermiculite has been used for a variety of products including loose-fill insulation, as a carrier for agricultural chemicals, and as an additive in potting soils.<sup>12</sup>

The vermiculite ore from Libby was mined from an open pit and beneficiated, crushed, sized, and sorted at the mine site into vermiculite concentrate. The vermiculite concentrate was shipped by rail to various exfoliation facilities around the United States, including the facility located in Spokane, Washington.

<sup>&</sup>lt;sup>4</sup> ATSDR, Toxicological Profile for Asbestos (updated), September 2001.

<sup>&</sup>lt;sup>5</sup> ATSDR, Asbestos, Year 2000 Medical Testing of Individuals Potentially Exposed to Asbestiform Minerals Associated with Vermiculite in Libby, Montana, A Report to the Community. August 23, 2001.

<sup>&</sup>lt;sup>6</sup> Fibrous Amphibole Contamination in Soil and Dust at Multiple Locations in Libby Poses an Imminent and Substantial Endangerment to Public Health, Christopher P. Weis, Ph. D. July 9, 2001.

<sup>&</sup>lt;sup>7</sup> W.R. Grace correspondence dated May 24, 1977.

<sup>8</sup> Robert S. Wright, Jerrold L. Abraham, Philip Harber, Bryan Burnett, Peter Morris, and Phil West. Fatal Asbestosis 50 years after Brief High Intensity Exposure in a Vermiculite Expansion Plant. American Journal of Respiratory Critical Care Medicine, Volume 165. Pp 1145-1148, 2002.

<sup>&</sup>lt;sup>9</sup> Pneumoconiosis in a Vermiculite End-Product User, Thomas Peter Howard, MD, US Veterans Administration, American Journal of Industrial Medicine, Volume 44, pages 214-217, 2003.

<sup>10</sup> Comba, P., Gianfagna, A., and Peoletti, L. Pleural mesothelioma Cases in Biancavilla are related to a New Fluoro-Edenite Fibrous Amphibole. Archives of Environmental Health. Volume 58, Number 4, pages 229 -232. April 2003.

ATSDR, Asbestos, Year 2000 Medical Testing of Individuals Potentially Exposed to Asbestiform Minerals Associated with Vermiculite in Libby, Montana, A Report to the Community. August 23, 2001.

<sup>&</sup>lt;sup>12</sup> Industrial Rocks and Minerals, 6th Edition, Society for Mining, Metallurgy, and Exploration, Inc. 1994.

## **Background**

Vermiculite products manufactured at the exfoliation plant in Spokane, Washington, from ore mined in Libby, Montana, were sold under the trade name Zonolite. In 2000, EPA tested samples from a bag of Zonolite Chemical Packaging Vermiculite and found it was contaminated with a

Figure 1. Commercial Vermiculite



significant quantity of amphibole asbestos.<sup>13</sup> While Zonolite Chemical Packaging Vermiculite is no longer being commercially produced, the product was still offered for sale at two Seattle area stores that sell garden supplies.

# History of the Former Vermiculite Plant in Spokane

The Spokane vermiculite facility was operated between 1951 and 1973 by two different companies, Vermiculite Northwest, Inc. and later W.R. Grace and Company. Both companies exfoliated vermiculite at the facility, and packaged and stored commercial vermiculite products on site. Records show up to 10,317 tons of vermiculite were processed at the site from January 1967 to October 1970.<sup>14</sup>

The vermiculite concentrate that was shipped to the Spokane site was off-loaded on the north side of the warehouse building. The concentrate was shoveled into a gas-fueled expansion furnace which heated the ore to approximately 1000° Fahrenheit (F). Water between the layered structure of the vermiculite ore converted to steam forcing the mineral itself to expand 6 to 30 times its original thickness. This expansion process is called exfoliation or popping. The exfoliated vermiculite went through a cooling chute and rock separator, after which it was put through a cyclone device to remove dust before the product was packaged. Dust from the vermiculite manufacturing process was generally swept off the floor, out the back door, and onto the ground on the north side of the warehouse. 16

After 1973, the site was occupied by the Wilburt Vault Company, which manufactured concrete products such as septic tanks and burial vaults. Spokane County acquired the site in January 2000 to store heavy equipment.<sup>17</sup>

### **Property Description**

The Spokane vermiculite facility is approximately two acres in size. It is located at 1318 N. Maple Street, Spokane, WA. The legal description of the property is Parcel B, portions of Block 23, 24 and 25 of the Chamberlains Addition. It is bordered by Maple Street to the west, Maxwell Avenue and an alley and private residences to the north, Cedar Street to the east, and Sharp Avenue, an alley, private residences, and Spokane County vehicle maintenance shops to the south.

During the period of study, a portion of the former warehouse stood on the southwest corner of the site. It has since been removed. A concrete slab, located east of the former warehouse, is where the vermiculite expansion furnace and vermiculite concentrate storage were situated. Several metal buildings, a concrete bridge abutment which used to connect with north Walnut Street, and a bluff, approximately 25 - 35 feet in elevation, are located north of the warehouse. Remnants of a railroad spur that serviced the site are located approximately 85 feet north of the warehouse running east to west.

<sup>&</sup>lt;sup>13</sup> U.S. Environmental Protection Agency (2000) Sampling and analysis of consumer garden products that contain vermiculite. EPA 744-R-00-010

 <sup>&</sup>lt;sup>14</sup> U. S. Environmental Protection Agency, Region 8, review of 103 Vermiculite Northwest, Inc. invoices for the period 1/14/67 to 10/14/70.
 <sup>15</sup> Interview on February 19, 2000 with Milton McDaniel, former supervisor at the Vermiculite Northwest / W.R. Grace vermiculite

exfoliation plant.

16 Interview on May 22, 2002 with Tim Groh, former employee at the Vermiculite Northwest / W.R. Grace vermiculite exfoliation plant.

17 U.S. Environmental Protection Agency, Region 10, Office of Environmental Assessment Report on Vermiculite Expansion Facilities

Investigation, May 23, 2000.
Spokane County Tax Records

<sup>&</sup>lt;sup>19</sup> Interview on May 22, 2002 with Tim Groh, former employee at the Vermiculite Northwest / W.R. Grace vermiculite exfoliation plant.

Figure2. Map of Spokane



## Previous Site Investigations

In 2000 and 2001, Ecology and Environment, Inc. (E&E), the Region 10 Office of Environmental Cleanup contractor, conducted two site visits that included sampling and analysis. During the first site visit, eight samples were taken. Analysis of the samples identified trace levels of asbestos in several samples and one sample with a

concentration of 2% asbestos.<sup>20</sup> During the second site visit, twenty-seven samples were collected from various locations. The analytical results showed trace levels in nine samples and two samples with concentrations of 2% and 3%.<sup>21</sup> The results were considered in determining sample locations for the OEA study.

<sup>20</sup> Trip Report, TDD: 00-03-0012. Ecology and Environment, Inc.

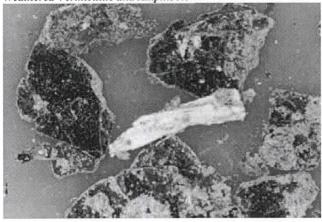
Vermiculite Northwest Removal Site Evaluation Trip Report, Spokane, Washington, TDD:01-07-0014. Ecology and Environment, Inc.

The following sections of this report detail the sampling techniques, analytical methods used in each phase of the study and results of analysis. The sampling techniques were developed by OEA and were detailed in a quality assurance project plan (QAPP) approved by chemists in the EPA Region 10 Quality Assurance and Data Unit. Modification of sampling procedures described in the QAPP were documented in the project notebook.

#### Phase I - General Surveillance of Site

EPA investigators reviewed available information and analytical data on the Spokane site to determine where samples should be taken. Once on site, accumulations of what appeared to be loose vermiculite concentrate or waste (stoner) rock were observed in the soil at several locations on the north and east side of the warehouse. The material observed was irregular platy mineral approximately 0.25 to 0.5 inches in diameter and black to dark grey in color. Several white colored fragments of a fibrous mineral were observed in the soils where the platy mineral was observed, as shown in Figure 3. Most of the platy mineral and white colored fragments were observed at depths between 7 and 14 inches below the soil surface. It was evident that the top layers of soil north and east of the warehouse were fill material. It also appeared that the dirt road surface east of the warehouse had been maintained with a road grader.

Figure 3. Weathered Vermiculite and Amphibole



In some areas under the fill material, OEA investigators observed a fine light brown to grey colored layer of material believed to be weathered ash from the 1980 eruption of Mount Saint Helens. Beneath the ash layer, on the north side of the warehouse, OEA investigators observed a layer of suspected vermiculite concentrate or stoner rock at a depth of approximately 14 inches.

#### Sampling Methods

On May 22 and 23, 2002, EPA investigators collected thirteen bulk soil samples from the site for analysis by polarized light microscopy (PLM). Most of the bulk samples consisted of approximately four ounces of soil taken from several locations where suspected vermiculite concentrate or stoner rock was observed.<sup>22</sup> Results of analysis of samples collected during the previous site visits by EPA contractors and other information provided during interviews with former W.R. Grace employees familiar with the site was also considered.

Three of the bulk soil samples were collected from under the warehouse building, either through the floor boards or through sections of a wall. Ten bulk soil samples were collected from locations around the north and east side of the warehouse, around the concrete bridge abutment, and across the hillside northeast of the warehouse. The soil samples were collected with pre-cleaned stainless steel spoons and were placed in four ounce glass containers with Teflon® lids.

#### **Analytical Methods**

Two different analytical techniques were used in this phase of the study. Analysis by PLM was used to determine if asbestos fibers were present in the bulk samples. X-Ray diffraction was used to more accurately determine the composition of the white colored fibrous mineral that was observed in the soils where suspected vermiculite concentrate or stoner rock was observed.

#### Polarized Light Microscopy (PLM) Analysis

The Phase I soil samples were analyzed by PLM by the Washington State Department of Ecology. The PLM analysis was intended to aid in the identification of areas where asbestos may be present. This analysis was not intended to quantify the amounts of asbestos present.

The method used was the Standard Operating Procedure for the Screening Analysis of Soil and Sediment Samples for Asbestos Content SOP: EIA-INGASED2.SOP dated 1/11/99.<sup>23</sup> The analysis involved placing aliquots of the soil samples into a test tube, adding water, then agitating. The contents of the test tube are poured through a 3-inch inside diameter 60 mesh, 250 micron (µm) sieve and the material left on the sieve is transferred to a plastic dish. Then the samples were dried and viewed with a stereo microscope to identify fiber bundles for analysis by PLM.

<sup>&</sup>lt;sup>22</sup> Waste material from the exfoliation process consisting of vermiculite ore that did not "pop".

<sup>23</sup> Method developed in Boston by Scott Clifford, a chemist with the Investigations and Analysis Unit, OEME U.S. EPA Region I.

Fiber bundles were mounted in 1.605 refractive index oil and analyzed using a Nikon Opti-phot Pol microscope at 200X magnification. Fibers observed were checked for diagnostic optical properties such as angle of extinction, sign of elongation, and refractive indices by central stop dispersion staining.<sup>24</sup>

#### X-Ray Diffraction (XRD) Analysis

Several fiber bundles, suspected to be Libby amphibole, were isolated from four bulk samples and washed with deionized water. The fiber bundles were hand picked for analysis by XRD with the aid of a stereo microscope. The majority of the fiber bundles appeared white, but some also appeared slightly green. When pressure was applied with steel forceps, the fiber bundles fragmented into long rigid fibers. Approximately one gram of fiber bundles was isolated from bulk soil samples 3, 4, 5, and 10 and combined into a clean glass vial and submitted for analysis.

XRD analysis was conducted by EPA staff at the Manchester Laboratory. A sample was disaggregated and lightly crushed under a hood using a mortar and pestle to reduce the grain size. Isopropanol was used to prevent modification of the crystal structure during crushing. An XRD mount was prepared using an eyedropper to place the isopropanol slurry with entrained specimen onto a quartz plate and allowed to dry. The XRD mount was also examined by optical microscopy.

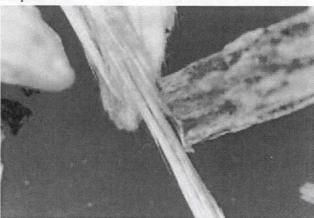
Mineral identification was conducted using EPA Region 10 Method XRD-QL for Compound Identification by X-ray Diffraction Analysis. Mineral identification was made by comparison with reference samples and with the Powder Diffraction File (PDF) maintained by the International Centre for Diffraction Data (ICDD).<sup>25</sup>

#### **Results of Analysis**

Eleven of the thirteen samples collected during Phase I were analyzed using PLM. Eight samples showed the presence of one or more types of asbestos, both amphibole and serpentine (chrysotile).

Fiber bundles, such as those shown in Figure 4, were isolated from soil samples and analyzed by XRD. The analysis of these fiber bundles revealed the diffraction pattern was typical of minerals belonging to the amphibole

Figure 4.
Bundles of Fibrous Amphibole Viewed at 20x Isolated from Soil Samples



group. The diffraction pattern was also compared to a fibrous sample of Libby amphibole mineral and was found to be similar. In addition to the amphiboles, a minor amount of vermiculite and trace amounts of biotite, talc, and calcite were detected in this sample.

Analysis of one health and safety sample obtained when specimens were collected from under the warehouse revealed an asbestos concentration of 0.024 f/cc of air.

## Phase II - Air Monitoring in an Experimental Enclosure

EPA investigators collected twelve larger specimens of soil that were co-located with twelve of the four-ounce bulk samples collected in Phase I. The larger Phase II specimens were needed for tests conducted in a sealed enclosure at the laboratory where air samples were taken.

#### Sampling Methods

Each specimen collected for Phase II consisted of approximately 1 cubic foot of soil with as much of the vegetation and larger stones removed as possible. The specimens were identified as Soil(s) 1 through 12, in Table 1 below. Ten were collected on May 22 and 23, 2002, and two were collected on July 16, 2002. Figure 5 shows the locations where the specimens were collected.

<sup>&</sup>lt;sup>24</sup> Susan Davis, Microscopist, Washington State Department of Ecology

<sup>&</sup>lt;sup>25</sup> Memo dated 1/30/03 from Dr. David Frank, EPA Region 10 Risk Assessment and Evaluation Unit.

Table 1.
Phase II - the Dates Collected and Locations for Soil Specimens 1-12.

Specimen Number	Date Collected	Location	
Soil #1	May 22 & 23, 2002	under warehouse	
Soil #2	May 22 & 23, 2002	under warehouse	
Soil #3	May 22 & 23, 2002	east of warehouse	
Soil #4	May 22 & 23, 2002	northeast of warehouse	
Soil #5	May 22 & 23, 2002	east of warehouse	
Soil #6	May 22 & 23, 2002	hillside west of bridge abutment	
Soil #7	May 22 & 23, 2002	hillside east of bridge abutment	
Soil #8	May 22 & 23, 2002	hillside east of bridge abutment	
Soil #9	May 22 & 23, 2002	hillside east of bridge abutment	
Soil #10	May 22 & 23, 2002	north of warehouse	
Soil #11	July 16, 2002	top of bluff	
Soil #12	July 16, 2002	railroad track bed	

Each soil specimen was placed inside clean stainless steel pans that were sealed closed for transport to the laboratory. Due to rainy conditions during sampling, the soil specimens were wet and not typical of the soil moisture content at this site during most of the year. To reduce the moisture content of the soil, the specimens were placed inside a drying oven for a period of 8-12 hours at 60° centigrade (C). After removal from the oven, they were allowed to cool for approximately 12 hours.

After drying, each specimen of soil was tested separately inside a sealed stainless steel Kewaunee Scientific Equipment (KSE) glove box. The inside dimensions of the glove box are approximately 4 feet long by 3 feet high with a depth of 21 inches at the top and 27 inches at the bottom (containing approximately 680 liters of air). The glove box is equipped with a front glass viewing panel and fluorescent lighting. The air inside the glove box was drawn through 25 millimeter (mm) air monitoring cassettes with 0.45  $\mu m$  mixed cellulose ester (MCE) filters. The cassettes were suspended inside the center of the glove box, approximately 14 inches above the work area, and were connected to two

Figure 5
Phase I and II Sample Locations for Soils 1-12. Aerial Photograph Courtesy of Spokane County.



Allegro® A-100 Hi Volume sampling pumps with R-3603 Tygon® tubing. Replacement air was introduced into the glove box during the sample pump operation by two sections of R-3603 Tygon® tubing. Metal clamps were closed over the tubing to keep air from escaping the glove box when the pumps were not operating.

Each specimen of soil was agitated with a stainless steel spoon for several minutes until airborne dust was visible. Air samples were collected from inside the work area approximately 1 hour after the soil was agitated. This allowed time for larger particles in the air to settle. This was done to reduce the risk of overloading the air monitoring filters which would allow for TEM analysis by direct transfer. Several of the air filters were observed under PCM to roughly estimate the degree of filter loading. A duplicate set of samples was collected using approximately the same air flow and sampling duration as the samples which appeared optimally loaded when viewed by PCM. The duplicate samples were shipped to Lab/Cor, Inc., a contract laboratory, for analysis by TEM.

Three types of air samples were taken for each specimen of soil studied. A work area background sample was collected by drawing air with a high volume pump through a pair of filters for approximately 30 minutes at a flow rate of 2.5 to 3.5 liters per minute, prior to putting the soil into the glove box. The samples collected during the soil agitation were collected by drawing air through a pair of filters for between 12 to 24 minutes at a flow rate of 2.5 to 3.5 liters per minute.

Between sampling events, while the work area inside the glove box was being cleaned, a health and safety sample was collected outside the glove box through a filter connected to a personal pump used by the investigator. The health and safety samples were collected for approximately 30 minutes with a high volume pump at a flow rate of 1.5 liters per minute. Health and safety samples are used to determine possible exposure to the staff using the glove box. The glove box was cleaned with a high efficiency particle arrestance (HEPA) vacuum and washed down with deionized water and wiped dry with clean paper towels.

Samples taken during disturbance of the first specimen of soil tested (Soil 1) were very time consuming to analyze due to the large number of fibers that needed to be counted. Based on the difficulty experienced with Soil 1, EPA investigators decided not to collect air samples using Soil 2.

#### **Analytical Methods**

#### Phase Contrast Microscopy (PCM)

Phase II samples were screened at the Manchester Environmental Laboratory (MEL) to determine appropriate particulate loading of the air filters. The method used was Asbestos by PCM NIOSH 7400.<sup>26</sup>

The filters were cut into wedges with a surgical steel knife and placed, particulate side up, on clean glass microscope slides. The slides were then placed into an aluminum hot block and 250 micro liters ( $\mu$ L) of acetone were injected into the inlet port of the hot block, collapsing the filter wedges. Then 3-5  $\mu$ L of triacetin was placed on the filter wedges to fix them to the slides and cover slips were applied. The slides were viewed with a Nikon phase contrast microscope (PCM) at 400X magnification. Fibers were sized using a Walton-Beckett graticule as reference. Only fibers greater than 5.0  $\mu$ m with a length to width (aspect) ratio<sup>27</sup> of greater than 3:1 were counted.<sup>28</sup> Fiber density is reported by MEL in fibers per square millimeter (f/mm²). This value has been converted to f/cc for this report.<sup>29</sup>

#### Transmission Electron Microscopy (TEM)

Phase II air monitoring samples were analyzed by Lab/ Cor using the NIOSH 7402 method for Asbestos by TEM.<sup>30</sup> In accordance with that analytical method, the sample filters were collapsed in acetone, coated with a thin layer of carbon at high vacuum, and placed on a 200 mesh copper TEM grid and allowed to dissolve in acetone until cleared of filter debris. The analysis was performed with a Philips 410 TEM equipped with an EDAX PV9800 X-ray analyzer. The samples were scanned at a magnification of approximately 990X using an accelerating voltage of 100 KV. The magnification was increased to about 10,000X for structure sizing. Fibers viewed using the NIOSH 7402 method are counted if they are greater than 5.0 µm in length with an aspect ratio greater than 3:1.<sup>31</sup> Fiber density is reported as f/cc.

## Results of Analysis

The Phase II data demonstrates that the disturbed soil emitted asbestos fibers under laboratory conditions. However, air sampling within an experimental enclosure may not be representative of real life exposures. This should be considered when comparing the data to regulatory standards.

<sup>&</sup>lt;sup>26</sup> Asbestos and other fibers by PCM Method 7400 NIOSH Manual of Analytical Methods, Fourth Edition, 8/15/94.

<sup>&</sup>lt;sup>27</sup> The 3:1 aspect ratio is also included in the EPA reference method published in 40 CFR Part 763.121 appendix A.

<sup>&</sup>lt;sup>28</sup> Susan Davis, Microscopist, Washington State Department of Ecology

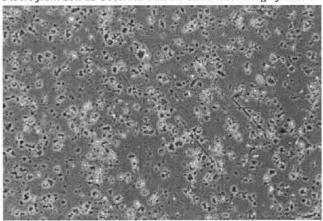
Fibers/cc =  $f/mm^2 x$  385  $mm^2$  divided by V (the average flow rate x sample duration) x 1000.

<sup>30</sup> Asbestos by TEM Method 7402 NIOSH Manual of Analytical Methods, Fourth Edition, 8/15/94.

<sup>31</sup> John Harris, Lab/Cor, Inc., Seattle, Washington

Analysis of air samples taken during disturbance of Soil 1 revealed the greatest variety and number of asbestos fibers. The majority of fibers were serpentine (chrysotile) although amphibole fibers were present as well. Chrysotile asbestos may have been added to some of the insulation products manufactured at this site.<sup>32</sup> The total concentration of asbestos fibers detected in duplicate samples was 10.461 f/cc of air and 10.713 f/cc.

Figure 6.
Fibers from Soil 12 Observed with a PCM at 400x Magnification



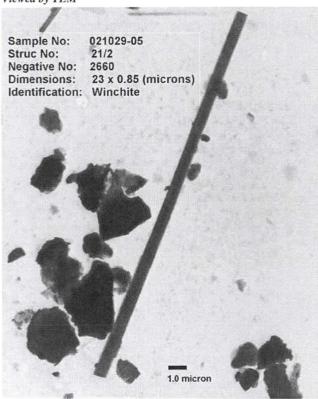
Analysis of samples taken during disturbance of Soil 3, 4, 10, and 12 revealed a number of fibers consisting mostly of amphibole asbestos. Total concentration of asbestos fibers detected in duplicate samples taken during disturbance of Soil 3 was 6.546 f/cc and 4.933 f/cc. Total concentration of asbestos fibers detected in duplicate samples taken during disturbance of Soil 4 was 4.818 f/cc and 5.624 f/cc. Total concentration of asbestos fibers detected in duplicate samples taken during disturbance of Soil 10 was 0.823 and 1.277 f/cc. The total concentration of asbestos fibers detected in duplicate samples taken during disturbance of Soil 12 was 1.526 f/cc and 2.186 f/cc.

Analysis of air samples taken during disturbance of Soils 5, 6, 7, and 11 revealed lower numbers of asbestos fibers. Total concentration of asbestos fibers detected in duplicate samples taken during disturbance of Soil 5 was 0.315 f/cc and 0.205 f/cc. Total concentration of asbestos fibers detected in duplicate samples taken during disturbance of Soil 6 was 0.153 f/cc and 0.158 f/cc. Total concentration of asbestos fibers detected in three samples taken during disturbance of Soil 7 was 0.096 f/cc, 0.136 f/cc, and 0.051 f/cc. The total concentration of asbestos fibers detected in duplicate samples taken during disturbance of Soil 11 was 0.262 f/cc and 0.238 f/cc.

Analysis of air samples taken during disturbance of Soil 8 revealed only one sample having numbers of asbestos fibers detected at a concentration of 0.051 f/cc. Both samples

resulting from disturbance of Soil 9 revealed no asbestos fibers detected. A summary of Phase II sampling and analysis are included in Table 2 on page 10.

Figure 7. Asbestos Fiber on Air Filter Derived from Soil 12 in Phase II Viewed by TEM



No asbestos fibers were detected in any of the work area background samples or the health and safety samples collected during Phase II of this study.

## Phase III - Task Based Monitoring On-Site

Phase III was intended to determine if asbestos could become airborne if workers on the site disturbed the soil. Two locations were selected for field exercises.

The first location, designated location C, was on top of the bluff on the north boundary of the site. This location was recommended by the ECL On-Scene Coordinator because of its close proximity to homes near the north side of the site. The second location, designated location A/B, was between the warehouse (Area A) and the rail bed (Area B). This location was selected because results of analysis for samples collected in Phase II indicated there was a significant concentration of amphibole fibers in the soil. Figure 8 is an aerial photograph showing the locations of Areas A, B, and C as well as the stationary monitoring locations.

<sup>32</sup> Interview on May 22, 2002 with Tim Groh, former employee at the Vermiculite Northwest / W.R. Grace vermiculite exfoliation plant.

Table 2.

Phase II - Airborne fiber concentrations associated with soil disturbance under experimental conditions.

Description	Sample Number	Sample collection Duration (D), Air Volume (AV)	Total Asbestiform Minerals by 7402 TEM f/cc (Libby amphiboles plus amosite and chrysotile)  10.461 10.713	
Soil 1 Under Warehouse GPS file Q052223a	02244010 02244011	D= 15 min, AV=2964 cc/min D= 15 min, AV= 3065 cc/min		
Soil 3 Concrete Pad GPS file Q05315b	02264106 02264107	D= 12 min, AV= 3034 cc/min D= 12 min, AV= 3051 cc/min	6.546 4.933	
Soil 4 Area A GPS file Q052223a	02244050 02244051	D= 12 min, AV= 3003 cc/min D= 12 min, AV= 3004 cc/min	4.818 5.624	
Soil 5 East edge of Concrete Pad GPS file Q052316a	02244056 02244057	D= 12 min, AV= 3032 cc/min D= 12 min, AV = 3033 cc/min	0.315 0.205	
Soil 6 West of Concrete Bridge Abutment GPS file Q052318a	02324660 02324661	D= 12 min, AV= 3123 cc/min D= 12 min, AV= 3012 cc/min	0.153 0.158	
Soil 7 Hillside GPS file Q052318b	02284404 02284405 02284406	D= 12 min, AV= 3310 cc/min D= 12 min, AV= 3323 cc/min D= 24 min, AV= 3164 cc/min	0.096 0.136 0.075	
Soil 8 Hillside GPS file Q052320a	02324654 02324655	D= 12 min, AV= 3140 cc/min D= 12min, AV= 3054 cc/min	0.051 ND*	
Soil 9 Hillside GPS file Q052320b	02274104 02274105	D= 12 min, AV= 3159 cc/min D= 12 min, AV= 3150 cc/min	ND ND	
Soil 10 Area A GPS file Q052315a	02274111 02274112	D= 12 min, AV= 3096 cc/min D= 12 min, AV= 3056 cc/min	0.823 1.277	
Soil 11 Area C GPS file Q082622b	Area C $02294305$ $D = 12 min, AV = 3051 cc/min$		0.262 0.238	
Soil 12 Area B GPS file Q082622a	02314604 02314605	D= 12 min, AV= 3132 cc/min D= 12 min, AV= 3125 cc/min	1.525 2.186	

 $<sup>*</sup>ND = None\ Detected$ 

During the sampling period, the wind direction and velocity fluctuated out of the north and west from 0 - 5 miles per hour (estimated). It was also noted the surface layer of soil at location C was somewhat dry but became more moist below a depth of three to four inches. Similarly, the soil below the gravel layer at location A/B was also moist.

#### Sampling Methods

On October 22 and 23, 2002, OEA investigators collected air samples from the site as part of the on-site monitoring phase of this study. Activities that disturbed the soil were conducted at two locations while air samples were collected with both stationary air monitors and personal air monitors.

A total of 32 samples was collected during Phase III. Of these, 12 were personal air monitoring samples for TEM analysis, 6 were personal air monitoring samples for PCM analysis, 7 were area monitoring samples (6 analyzed by TEM and 1 analyzed by PCM) and 7 samples were taken for quality assurance background and blank samples. To allow for some comparison of results between TEM and PCM analytical techniques, 5 samples were analyzed using both PCM and TEM.

At monitoring location C, investigators performed property maintenance tasks such as mowing, shoveling, raking, tilling, and leaf blowing. Two stationary monitoring stations were set up, one the west side and another on the east side of the location C.

Figure 8
Phase III Monitoring Areas A, B, and C. Aerial Photograph Courtesy of Spokane County.



At monitoring location A/B, a trench was excavated from east to west along the south side of the rail bed and two trenches along the north side of the warehouse. The trenches were excavated using a mid-size Kubota tractor equipped with a back-hoe, shown in Figure 9. The trenches were between 10 and 15 feet in length and approximately 2 to 3 feet in depth and approximately 1.5 and 2 feet wide. A leaf blower was used to simulate wind during this activity, and was also helpful in drying out damp soil.

The ambient air monitored at locations C and A/B was drawn through 25 millimeter (mm) air monitoring cassettes with 0.45  $\mu m$  mixed cellulose ester (MCE) filters. The cassettes were suspended approximately 4 feet above the ground and were connected to Allegro®A-100 Hi Volume sampling pumps with R-3603 Tygon® tubing. The pumps were calibrated before and after the sampling using a Gilibrator® electronic micro processor controlled calibration unit equipped with high flow cell capable of measuring 2 to 30 liters of air/minute.

Figure 9.
Task Based Monitoring in Phase III Excavation at Area A/B



One EPA worker operated the power equipment while observers roamed inside the locations and around the perimeter. The worker operating the power equipment wore two personal Gilian HFS-513 air sampling pumps connected with R-3603 Tygon® tubing to either 0.45  $\mu m$  MCE filters for TEM analysis or 0.8  $\,m$  MCE filters for PCM analysis. The observers wore a single Gilian HFS-513 air sampling pump connected with R-3603 Tygon® tubing to a 0.45  $\mu m$  MCE filter for TEM analysis. The personal monitoring pumps were calibrated before and after the sampling using a Gilibrator® electronic micro processor controlled calibration unit equipped with a standard flow cell capable of measuring 20 cc to 6 liters of air per minutes.

#### **Analytical Methods**

Air filter samples collected during Phase III were analyzed using PCM and TEM. The personal monitoring samples collected during Phase III were analyzed by Lab/ Cor using the NIOSH 7402 method. Stationary monitoring samples were analyzed using the ISO 10312 method for direct identification of asbestos in airborne samples.<sup>33</sup> This analysis is similar to the NIOSH 7402 method. Generally

the fibers are counted if they are greater than 5.0  $\mu m$  in length with an aspect ratio greater than 5:1. The ISO 10312 method also allows for counting PCM equivalent fibers with an aspect ratio of 3:1 or greater, which are longer than 5.0  $\mu m$  and with a diameter between 0.2 and 3.0  $\mu m$ .<sup>34</sup> Fiber density is reported as f/cc.

#### **Results of Analysis**

Samples derived from personal monitors at Location C were analyzed using either PCM, TEM, or both. Three samples from Location C analyzed by PCM contained fibers with concentrations ranging from 0.11 f/cc to 0.25 f/cc. Six samples from Location C analyzed by TEM contained no asbestos fibers. (See Table 3) Two air samples were collected using area monitors in Location C. These monitors were located at the east and west ends of the work area. Both samples were analyzed by TEM. The east stationary monitor contained one chrysotile fiber and three amphibole fibers. The west stationary monitor contained one chrysotile fiber.

At location A/B three samples from the investigators' personal monitors that were analyzed by TEM contained

Table 3.

Phase III - Personal Monitor Sample Analysis Results showing levels detected by PCM and TEM

(9 detects of 18 personal monitoring samples collected)

Description	Sample No.	Location	Activity	Sample collection - Duration (D) - Air Volume (AV)	PCM fibers/cc	7402 TEM fibers/cc
Soil #11	02434357	Area C on top of bluff	Observer Equipment operator - roto till	D= 60 minutes AV= 1.5 liters/minute	0.25	NA
Soil #11	02434363	Area C on top of bluff	Equipment operator - leaf blower	D= 60 minutes AV= 2.5 liters/minute	0.11	ND
Soil #11	02434366	Area C on top of bluff	Observer	D= 60 minutes AV= 2.3 liters/minute	0.16	ND
Soil #12	02434370	Area A/B - rail bed	Equipment operator - leaf blower	D= 40 minutes AV= 2.5 liters/minute	NA	0.010
Soil #12	02434372	Area A/B - rail bed	Observer	D= 40 minutes AV= 2.2 liters/minute	0.02	ND
Soil # 4, 10	02434375	Area A/B - north of warehouse	Observer	D= 72 minutes AV= 2.4 liters/minute	NA	0.033
Soil # 4, 10	02434376	Area A/B - north of warehouse	Equipment operator - leaf blower	D= 71 minutes AV= 1.5 liters/minute	0.16	0.045
Soil # 4, 10	02434377	Area A/B - north of warehouse	Observer	D= 45 minutes AV= 1.5 liters/minute	NA	ND
Soil # 4, 10	02434379	Area A/B - north of warehouse	Equipment operator - backhoe	D= 72 minutes AV= 2.4 liters/minute	0.04	NA

ND = None Detected NA = Not Analyzed

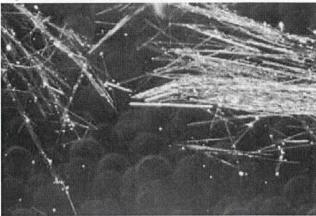
<sup>33</sup> Ambient Air-Determination of Asbestos Fibres-Direct-Transfer Transmission Electron Microscopy Method. ISO 10312, First edition 1995-05-01.

<sup>34</sup> John Harris, Lab/Cor, Inc., Seattle, Washington

amphibole fibers. The asbestos fiber concentrations ranged from 0.01 f/cc to 0.045 f/cc. Three air samples from personal monitors analyzed by PCM contained fibers with concentrations ranging from 0.02 f/cc to 0.045 f/cc. The air sample collected from the area A/B east stationary monitor analyzed by TEM contained three amphibole fibers. A summary of the Phase III analysis of personal monitor samples is included in Table 3.

Phase III results demonstrate that if disturbed, soil at all three locations will emit asbestos fibers. By performing simulated property maintenance and excavation tasks, investigators were able to release measurable amounts of asbestos fibers that were captured in some of the area monitors down wind of the activities or in some of the personal monitors worn by EPA workers.

Figure 10.
Bundles of Fibrous Amphibole Viewed at 200x by PLM
Dispersion Staining Objective in 1.605 Refractive Index Liquid



## Fiber Morphology

During the OEA study the majority of asbestos observed appeared fibrous. Images captured using various microscopic techniques (P.M., PCM, TEM, and stereo-microscope) clearly show asbestos fibers and/or fiber bundles in several samples. (See figures 2, 3, 4, 5, and 7) Approximately 52% of the total asbestos fibers counted by TEM during Phase II and III had an aspect ratio of 20:1. Approximately 15% of

the fibers counted were greater than or equal to 20  $\mu m$  in average length. The analytical methods used in this study did not count fibers less than 5  $\mu m$  in average length and less than 0.2  $\mu m$  in average width. It is important to note that shorter thin fibers less than 5.0  $\mu m$  in length may have toxicological significance.

## **Risk Analysis**

Data from analysis of samples collected at this site during Phases II and III were used by OEA to perform a risk analysis for potential future on-site residents and workers. This analysis does not address residents that currently live adjacent to the site because no samples were collected from their homes or yards.

Using standard conservative assumptions about inhalation rates, exposure frequency and exposure duration, the data suggest elevated risks to on-site workers or residents. The

physical characteristics (length and diameter) of the Libby amphiboles identified in study samples analyzed likely contribute to greater toxicological potency and therefore greater risk than reflected in EPA's current inhalation unit risk value.<sup>35</sup>

While the risk of exposure to Libby amphiboles from this site may be minimized if the soil is left undisturbed, the potential for risk of exposure would likely increase if contaminated soil is disturbed enough to suspend asbestos fibers in air through excavation or wind erosion.

## Summary

The OEA study of the former vermiculite processing facility in Spokane, Washington, revealed that asbestos in the soil can become airborne if the soil is disturbed.

Even when the asbestos concentrations in the soil appear very low by PLM, agitation of asbestos contaminated soil in a laboratory enclosure resulted in a very high concentration of airborne fibers. During on-site excavation and property maintenance activities, air monitoring revealed the concentration of asbestos fibers at levels which suggest an elevated risk.

Therefore, the asbestos contaminated soil at this site may pose a potential health hazard for workers involved in future excavation and development of the property

<sup>35</sup> Memo regarding Vermiculite Expansion Plant, Spokane, WA, Preliminary Risk Assessment, Julie Wroble, Toxicologist, U.S. Environmental Protection Agency, Region 10, May 29, 2003.